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	1	(11) International Publication Number: WO 99/45085
C10G 67/04, 45/64	A1	(43) International Publication Date: 10 September 1999 (10.09.99
21) International Application Number: PCT/U 22) International Filing Date: 29 January 1999	S99/021:	BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GI
30) Priority Data: 60/077,070 6 March 1998 (06.03.98) 09/107,835 30 June 1998 (30.06.98)	τ	SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM European patent (AT, BE, CH, CY, DE, DK, ES, FI, FI GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (B)
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(54) Title: PREPARING A HIGH VISCOSITY INDEX, LOW BRANCH INDEX DEWAXED OIL

(57) Abstract

An integrated process for preparing a lubricating oil base stock includes an isomerization step followed by a solvent dewaxing step. A waxy feed is isomerized to an intermediate pour point, which is at least 6 °C above a target pour point, over a select molecular sieve having specified pore properties. The isomerized oil is then solvent dewaxed to a very low pour point. This process produces a dewaxed lubricating oil base stock which has an exceptionally high viscosity index.

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Synthetic lubricants may be used when very low pour point and very 1 high viscosity index lubricants are desired. But the starting materials used to 2 make the synthetic lubricants, and the processes used in manufacturing these 3 lubricants, are very expensive. The need remains for a lubricating oil base 4 stock, having synthetic-like properties but prepared from a mineral oil feed 5 using methods which are similar to those presently employed in refinery 6 7 processes. A major breakthrough came with the discovery of new dewaxing 8 catalysts which were found to isomerize rather than crack the wax molecules. 9 Isomerization alters the molecular structure of wax molecules, and generally 10 decreases the pour point of the molecule without significantly changing its 11 boiling point. In contrast to solvent dewaxing and to wax cracking, isomerized 12 molecules are retained in the lubricating oil base stock, increasing yield of 13 14 lubricating oil base stock without reducing viscosity index significantly. A particularly important group of isomerization catalysts include the 15 silicoaluminophosphate molecular sieves (SAPO). The preparation of 16 silicoaluminophosphate molecular sieves, including SAPO-11, SAPO-31 and 17 SAPO-41, are taught, for example, in U.S. Patent No. 4,440,871. Dewaxing 18 processes using such molecular sieves are taught in U.S. Patent 19 No. 4,859,311; U.S. Patent No. 4,867,862; U.S. Patent No. 4,921,594; U.S. 20 Patent No. 5,082,986; U.S. Patent No. 5,135,638; U.S. Patent No. 5,149,421; 21 U.S. Patent No. 5,246,566; U.S. Patent No. 5,413,695; and U.S. Patent 22 23 No. 4,960,504. SAPO molecular sieves belong to an important class of non-zeolitic 24 molecular sieve dewaxing catalysts which are useful as isomerization 25 catalysts for converting wax and wax-like components. Non-zeolitic molecular 26 sieves are microporous compositions that are formed from AlO₂ and PO₂ 27 tetrahedra which form 3-dimensional crystalline structures, and are described 28

broadly for this use in U.S. Patent No. 4,906,351 and U.S. Patent

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at least 200:1. WO 96/13563 teaches an isomerization process for producing a high viscosity index lubricant using a low acidity large pore molecular sieve having a crystal size of less than 0.1 micron, an alpha value of not more than 30 and containing a noble metal hydrogenation component. EP 225053 teaches isomerization dewaxing using a large pore, high silica zeolite dewaxing catalyst, followed by a subsequent dewaxing step which selectively removes the more waxy n-paraffin components. The selective dewaxing step may be either a solvent or a catalyst dewaxing, preferably using highly shape

selective zeolite such as ZSM-22 or ZSM-23.

 While the intermediate pore size molecular sieves have been shown to be effective for producing high viscosity index lubricating oil base stocks, the need remains for even higher viscosity index products which have been dewaxed to a low pour point.

SUMMARY OF THE INVENTION

An object of the present invention is to provide a process for producing an oil, having a very high viscosity index and a very low pour point, which is suitable for use as a lubricating oil base stock. The feedstock to the present process is a waxy feed which may be derived from mineral oils and mineral oil crudes. The oil which is produced has lubricating oil properties that approach, and may exceed, the lubricating oil properties of a synthetic lubricating oil base stock. Accordingly, the present invention provides a process for preparing an oil suitable for use as a lubricating oil base stock and having a viscosity index of greater than 140 and a target pour point of less than or equal to –10°C comprising:

a) contacting a waxy feed over a catalyst comprising a molecular sieve having 1-D pores with a pore diameter of between about 5.0 Å and about 7.0 Å, and at least one Group VIII metal, at a pressure of from about 15 psig (103 kPa) to about 2500 psig (13.8 MPa) to produce an isomerized oil having a pour point of at least 6°C above a target pour point; and

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DETAILED DESCRIPTION OF THE INVENTION

Normal paraffins are a major contributor to wax and a high pour point in a lubricating oil base stock. It is desirable to isomerize the normal paraffins to low pour point branched paraffins which retain the boiling range of the normal paraffins from which there were converted.

Among other factors, the present invention is based on the discovery that the number of branches produced while isomerizing a normal paraffin molecule significantly impacts the quality of the dewaxed oil product. For example, isomerizing a normal C₂₄ paraffin, tetracosane, using a large pore zeolite catalyst conventionally taught for wax isomerization, generally produces a significant quantity of triply branched paraffin isomers. Even medium pore catalysts taught for wax isomerization, when isomerizing a waxy feed to a low pour point, produces significant quantities of the triply branched isomers. While not wishing to be bound by theory, it is believed that normal paraffins are first converted during wax isomerization to a singly branched paraffin having a methyl (--CH₃) or ethyl (--C₂H₅), branch near the end of the paraffin backbone. Additional isomerization reactions move the branch toward the center of the paraffin molecule and/or add a second branch to the paraffin molecule. Each of these two isomerization reaction steps reduces pour point.

However, conventional single stage and/or large pore zeolite dewaxing processes are unselective for forming branches. These unselective catalysts produce triply (or even more highly) branched isomers along with the singly and doubly branched isomers before reaching the target pour point. These highly branched molecules have an increased tendency to crack and have a lower viscosity index than do singly or doubly branched paraffins. Furthermore, the addition of a third branch to a doubly branched paraffin often results in relatively little additional pour point reduction. Thus, these conventional processes are prevented from producing lubes with the desired viscosity index and pour point properties.

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stocks of this kind and also of other distillate fractions since they normally
contain significant amounts of waxy n-paraffins.

As used herein, the term "waxy feed" includes petroleum waxes. 5 Exemplary suitable feeds for use in the process of the invention also include 6 waxy distillate stocks such as gas oils, lubricating oil stocks, synthetic oils and 7 waxes such as those by Fischer-Tropsch synthesis, high pour point 8 polyalphaolefins, foots oils, normal alpha olefin waxes, slack waxes, deoiled 9 waxes and microcrystalline waxes. Slack wax is wax recovered from a 10 conventional solvent dewaxing process. Slack wax can be obtained from 11 either a straight run gas oil, a hydrocracked lube oil or a solvent refined lube 12 oil. Hydrocracking is preferred because that process can also reduce the 13 nitrogen content to low values. With slack wax derived from solvent refined 14 oils, deoiling can be used to reduce the nitrogen content. Optionally, 15 hydrotreating of the slack wax can be carried out to lower the nitrogen content 16 thereof. Slack waxes possess a very high viscosity index, normally in the 17 range of from 120 to 200, depending on the oil content and the starting 18 material from which the wax has been prepared. Slack waxes are therefore 19 20 eminently suitable for the preparation of lubricating oils having very high viscosity indices, i.e., from about 140 to about 180. Foots oil is prepared by 21 separating oil from the wax. The isolated oil is referred to as foots oil. 22

The feedstock employed in the process of the invention preferably contains greater than about 50% wax, more preferably greater than about 80% wax, most preferably greater than about 90% wax. However, a highly paraffinic feed having a high pour point, generally above about 0°C, more usually above about 10°C, but containing less than 50% wax is also suitable for use in the process of the invention. Such a feed should preferably contain greater than about 70% paraffinic carbon, more preferably greater than about 80% paraffinic carbon, most preferably greater than about 90% paraffinic carbon.

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molecular sieves having such effective pore sizes are advantageously usable in the practice of the present invention. Indeed, it is essential that the intermediate pore size molecular sieve catalysts used in the practice of the present invention have a very specific pore shape and size as measured by X-ray crystallography. First, the intracrystalline channels must be parallel and must not be interconnected. Such channels are conventionally referred to as 1-D diffusion types or more shortly as 1-D pores. The classification of intrazeolite channels as 1-D, 2-D and 3-D is set forth by R. M. Barrer in Zeolites, Science and Technology, edited by F. R. Rodrigues, L. D. Rollman and C. Naccache, NATO ASI Series, 1984 which classification is incorporated in its entirety by reference (see particularly page 75). Known 1-D zeolites include cancrinite hydrate, laumontite, mazzite, mordenite and

zeolite L.

In general, the pores of the molecular sieve have a major axis between about 5.0 Å and about 7.0 Å, i.e. the pore diameter of the molecular sieve is between about 5.0 Å and about 7.0 Å. In one embodiment, the preferred molecular sieves useful in the practice of the present invention have pores which are oval in shape, by which is meant the pores exhibit two unequal axes referred to herein as a minor axis and a major axis. The term oval as used herein is not meant to require a specific oval or elliptical shape but rather to refer to the pores exhibiting two unequal axes. In particular, the 1-D pores of the preferred molecular sieves useful in the practice of the present invention have a minor axis between about 3.9Å and about 4.8Å and a major axis between about 5.4Å and about 7.0Å as determined by conventional X-ray crystallography measurements, following the measurement convention of W. M. Meier and D. H. Olson, *ATLAS OF ZEOLITE STRUCTURE TYPES*, Butterworth-Heinemann, Third Revised Edition, 1992.

The present invention makes use of molecular sieve catalysts with selected shape selectivity properties. These shape selectivity properties are defined by carrying out standard isomerization selectivity tests for isomerizing tetracosane (n-C₂₄). The test conditions include a total pressure of 1000 psig

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4. Quantify the relative frequency of branch occurrence by comparing the integrated intensity of its terminal methyl carbon to the intensity of a single carbon (=total integral/number of carbons per molecule in the mixture). For the unique case of the isopropyl branch, where both methyl occur at the same resonance position, the intensity was divided by two before doing the frequency of branch occurrence calculation.

All measurements were performed with Varian 300 MHz spectrometers. In all cases the spectral width was limited to the saturated carbon region, about 0-80 ppm vs. TMS (tetramethyl silane). 15-25% solutions by weight in chloroform-d1 excited by 45° pulses followed by an 0.8 sec acquisition time. In order to minimize non uniform intensity data, the proton decoupler was gated off during a 10 sec delay prior to the excitation pulse and on during acquisition. Total experiment times ranged from 11-80 minutes. The DEPT and APT sequences were carried out according to literature descriptions with minor deviations described in the Varian operating manuals.

A catalyst, if it is to qualify as a catalyst of this invention, when tested in this manner, must convert sufficient normal C₂₄ paraffin to form an isomerized product having a pour point of about –15°C or less and a branching index of less than about 1.75. Non-zeolitic molecular sieves having the characteristics of an intermediate pore size molecular sieve as described herein are useful in the present process. Non-zeolitic molecular sieves are microporous compositions that are formed from AlO₂ and PO₂ tetrahedra. Thus, the process of the invention may be carried out using a catalyst comprising an intermediate pore size non-zeolitic molecular sieve and at least one Group VIII metal. Non-zeolitic molecular sieves are described, for example, in U.S. Patent No. 4,861,743, the disclosure of which is completely incorporated herein by reference for all purposes. Non-zeolitic molecular sieves include aluminophosphates (AlPO₄) as described in U.S. Patent No. 4,310,440, silicoaluminophosphates (SAPO), metalloaluminophosphates

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1 intermediate pore size silicoaluminophosphate molecular sieve for use in the

- 2 process of the invention is SAPO-11. When combined with a platinum or
- 3 palladium hydrogenation component, the SAPO-11 converts the waxy
- 4 components to produce a lubricating oil having excellent yield, very low pour
- 5 point, low viscosity and high viscosity index.
- 6 SAPO-11 comprises a silicoaluminophosphate material having a
- 7 three-dimensional microporous crystal framework structure of PO₂, AlO₂ and
- 8 SiO₂ tetrahedral units whose unit empirical formula on an anhydrous basis is:
- 9 mR: $(Si_xAI_vP_z)O_2$
- 10 wherein "R" represents at least one organic templating agent present in the
- intracrystalline pore system; "m" represents the moles of "R" present per mole
- of (Si_xAl_yP_z)O₂ and has a value of from zero to about 0.3, "x", "y" and "z"
- 13 represent respectively, the mole fractions of silicon, aluminum and
- phosphorous, wherein "x" has a value greater than zero (0), and "y" and "z"
- each have a value of at least 0.01. The silicoaluminophosphate has a
- 16 characteristic X-ray powder diffraction pattern which contains at least the
- 17 d-spacings (as-synthesized and calcined) set forth below in Table I. When
- 18 SAPO-11 is in the as-synthesized form, "m" preferably has a value of from
- 19 0.02 to 0.3.

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20		TABLE I	
21 22 <u>2Θ</u>		Interplanar d-spacings (Å)	Relative <u>Intensity, I/I_o</u>
23	9.4-9.65	9.41-9.17	m
24	20.3-20.6	4.37-4.31	m
25	21.0-21.3	4.23-4.17	vs
2 6	22.1-22.35	4.02-3.99	m
27	22.5-22.9 (doublet)	3.95-3.92	m-s

The most particularly preferred intermediate pore SAPO prepared by the present process is SM-3, which has a crystalline structure falling within that of the SAPO-11 molecular sieves. The preparation of SM-3 and its unique characteristics are described in U.S. Patent Nos. 4,943,424 and

intermediate pore size silicoaluminophosphate molecular sieve for use in the process of the invention is SAPO-11. When combined with a platinum or

- 3 palladium hydrogenation component, the SAPO-11 converts the waxy
- 4 components to produce a lubricating oil having excellent yield, very low pour
- 5 point, low viscosity and high viscosity index.

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0.02 to 0.3.

SAPO-11 comprises a silicoaluminophosphate material having a three-dimensional microporous crystal framework structure of PO₂, AlO₂ and SiO₂ tetrahedral units whose unit empirical formula on an anhydrous basis is:

9 mR: $(Si_xAl_yP_z)O_2$

wherein "R" represents at least one organic templating agent present in the 10 intracrystalline pore system; "m" represents the moles of "R" present per mole 11 of (Si_xAl_yP_z)O₂ and has a value of from zero to about 0.3, "x", "y" and "z" 12 represent respectively, the mole fractions of silicon, aluminum and 13 phosphorous, wherein "x" has a value greater than zero (0), and "y" and "z" 14 each have a value of at least 0.01. The silicoaluminophosphate has a 15 characteristic X-ray powder diffraction pattern which contains at least the 16 d-spacings (as-synthesized and calcined) set forth below in Table I. When 17 SAPO-11 is in the as-synthesized form, "m" preferably has a value of from 18

20		TABLE I	
21 22	<u>2Θ</u>	Interplanar d-spacings (Å)	Relative Intensity, I/I _o
23	9.4-9.65	9.41-9.17	m
24	20.3-20.6	4.37-4.31	m
25	21.0-21.3	4.23-4.17	vs
26	22.1-22.35	4.02-3.99	m
27	22.5-22.9 (doublet)	3.95-3.92	m-s

The most particularly preferred intermediate pore SAPO prepared by the present process is SM-3, which has a crystalline structure falling within that of the SAPO-11 molecular sieves. The preparation of SM-3 and its unique characteristics are described in U.S. Patent Nos. 4,943,424 and

of $(Si_xAl_yP_z)O_2$ and has a value of from zero to 0.3; "x", "y" and "z" represent,

- 2 respectively, the mole fractions of silicon, aluminum and phosphorous,
- 3 wherein "x" has a value greater than zero (0), and "y" and "z" each have a
- 4 value of at least 0.01. SAPO-41 has characteristic X-ray powder diffraction
- 5 pattern (as-synthesized and calcined) which contains at least the d-spacings
- 6 set forth below in Table III. When SAPO-41 is in the as-synthesized form,
- 7 "m" preferably has a value of from 0.02 to 0.03.

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8	TABLE III			
9 10	<u>2Θ</u>	Interplanar d-spacings (Å)	Relative Intensity, I/I _o	
11	13.6-13.8	6.51-6.42	w-m	
12	20.5-20.6	4.33-4.31	w-m	
13	21.1-21.3	4.21-4.17	vs	
14	22.1-22.3	4.02-3.99	m-s	
15	22.8-23.0	3.90-3.86	m	
16	23.1-23.4	3.82-3.80	w-m	
17	25.5-25.9	3.493-3.44	w-m	

The group of intermediate pore size zeolites useful in the present process include ZSM-22, ZSM-23, ZSM-35, ZSM-48 and SSZ-32. These catalysts are generally considered to be intermediate pore size catalysts based on the measure of their internal structure as represented by their Constraint Index. Zeolites which provide highly restricted access to and egress from their internal structure have a high value for the Constraint Index, while zeolites which provide relatively free access to the internal zeolite structure have a low value for their Constraint Index. The method for determining Constraint Index is described fully in U.S. Pat. No. 4,016,218 which is incorporated herein by reference.

One of the zeolites of the present invention, ZSM-22, is a highly siliceous material which includes crystalline three-dimensional continuous framework silicon containing structures or crystals which result when all the oxygen atoms in the tetrahedra are mutually shared between tetrahedral

of (Si_xAl_yP_z)O₂ and has a value of from zero to 0.3; "x", "y" and "z" represent,

- 2 respectively, the mole fractions of silicon, aluminum and phosphorous,
- 3 wherein "x" has a value greater than zero (0), and "y" and "z" each have a
- 4 value of at least 0.01. SAPO-41 has characteristic X-ray powder diffraction
- 5 pattern (as-synthesized and calcined) which contains at least the d-spacings
- 6 set forth below in Table III. When SAPO-41 is in the as-synthesized form,
- 7 "m" preferably has a value of from 0.02 to 0.03.

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8	TABLE III			
9 10	<u>20</u>	Interplanar d-spacings (Å)	Relative Intensity, I/I _o	
11	13.6-13.8	6.51-6.42	w-m	
12	20.5-20.6	4.33-4.31	w-m	
13	21.1-21.3	4.21-4.17	vs	
14	22.1-22.3	4.02-3.99	m-s	
15	22.8-23.0	3.90-3.86	m	
16	23.1-23.4	3.82-3.80	w-m	
17	25.5-25.9	3.493-3.44	w-m	

The group of intermediate pore size zeolites useful in the present process include ZSM-22, ZSM-23, ZSM-35, ZSM-48 and SSZ-32. These catalysts are generally considered to be intermediate pore size catalysts based on the measure of their internal structure as represented by their Constraint Index. Zeolites which provide highly restricted access to and egress from their internal structure have a high value for the Constraint Index, while zeolites which provide relatively free access to the internal zeolite structure have a low value for their Constraint Index. The method for determining Constraint Index is described fully in U.S. Pat. No. 4,016,218 which is incorporated herein by reference.

One of the zeolites of the present invention, ZSM-22, is a highly siliceous material which includes crystalline three-dimensional continuous framework silicon containing structures or crystals which result when all the oxygen atoms in the tetrahedra are mutually shared between tetrahedral

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replacement ions are those taken from Group VIII of the Periodic Table, 1 especially platinum, palladium, iridium, osmium, rhodium and ruthenium. 2

ZSM-22 freely sorbs normal hexane and has a pore dimension greater 3 than about 4Å. In addition, the structure of the zeolite provides constrained 4 access to larger molecules. The Constraint Index as determined by the 5 procedure set forth in U.S. Pat. No. 4,016,246 for ZSM-22 has been 6 determined to be from about 2.5 to about 3.0. 7

Another zeolite which can be used with the present invention is the synthetic crystalline aluminosilicate referred to as ZSM-23, disclosed in U.S. Pat. No. 4,076,842, the contents of which are incorporated herein by reference. The ZSM-23 composition has a characteristic X-ray diffraction pattern as set forth herein in Table V. 12

13	TA	TABLE V				
14 15	Interplanar d-spacings (Å)	Relative Intensity, I/I _o				
16	11.2 ±0.23	m				
17	10.1 ±0.20	W				
18	7.87 ±0.15	W				
19	5.59 ±0.10	W				
20	5.44 ±0.10	W				
21	4.90 ±0.10	W				
22	4.53 ±0.10	S				
23	3.90 ± 0.08	VS				
24	3.72 ±0.08	VS				
25	3.62 ±0.07	VS				
26	3.54 ±0.07	m				
27	3.44 ±0.07	S				
28	3.36 ±0.07	W				
29	3.16 ±0.07	W				
30	3.05 ±0.06	W				
31	2.99 ±0.06	w				
32	2.85 ±0.06	W				
33	2.54 ±0.05	m				
34	2.47 ±0.05	W				
35	2.40 ±0.05	W				
36	2.34 ±0.05	W				
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1 replacement ions are those taken from Group VIII of the Periodic Table,

2 especially platinum, palladium, iridium, osmium, rhodium and ruthenium.

ZSM-22 freely sorbs normal hexane and has a pore dimension greater than about 4Å. In addition, the structure of the zeolite provides constrained access to larger molecules. The Constraint Index as determined by the procedure set forth in U.S. Pat. No. 4,016,246 for ZSM-22 has been determined to be from about 2.5 to about 3.0.

Another zeolite which can be used with the present invention is the synthetic crystalline aluminosilicate referred to as ZSM-23, disclosed in U.S. Pat. No. 4,076,842, the contents of which are incorporated herein by reference. The ZSM-23 composition has a characteristic X-ray diffraction pattern as set forth herein in Table V.

13	TA	BLE V
14 15	Interplanar <u>d-spacings (Å)</u>	Relative <u>Intensity, I/I_o</u>
15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30	d-spacings (Å) 11.2 ±0.23 10.1 ±0.20 7.87 ±0.15 5.59 ±0.10 5.44 ±0.10 4.90 ±0.10 4.53 ±0.10 3.90 ±0.08 3.72 ±0.08 3.62 ±0.07 3.54 ±0.07 3.44 ±0.07 3.16 ±0.07 3.05 ±0.06	Intensity, I/I _o m w w w w w s vs vs vs ws w w
31 32 33 34 35 36 37	2.99 ±0.06 2.85 ±0.06 2.54 ±0.05 2.47 ±0.05 2.40 ±0.05 2.34 ±0.05	w w m w w

synthesized and in the anhydrous state, in terms of mole ratios of oxides, is as follows:

- 3 (0.05-2.0)R₂O:(0.1-2.0)M₂O:Al₂O₃:(20-less than 40)SiO₂
- 4 where M is an alkali metal cation and R is an organic nitrogen-containing
- 5 cation, such as an N-lower alkyl-N-N'-isopropyl-imidazolium cation. SSZ-32
- 6 has a mole ratio of silicon oxide to aluminum oxide in the range of 20 to less
- 7 than 40, and has essentially the same X-ray diffraction pattern of ZSM-23.
- 8 Hydroconversion processes using SSZ-32 are disclosed, for example, in U.S.
- 9 Patent Nos. 5,300,210 and in 5,397,454.

ZSM-48 is a crystalline aluminosilicate zeolite which is suitable as a dewaxing catalyst for the present invention. Zeolite ZSM-48 is disclosed in U.S. Patent No. 4,585,747, the entire disclosure of which is incorporated herein by reference for all purposes, and has a characteristic X-ray diffraction pattern as set forth in Table VI.

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	Table VI		
Interplanar	Relative		
d-spacings (Å)	Intensity, I/I _o		
11.8 ± 0.2	S		
10.2 ± 0.2	w-m		
7.2 ± 0.15	W		
4.2 ± 0.08	VS		
3.9 ± 0.08	VS		
3.6 ± 0.06	W		
3.1 ± 0.05	W		
2.85 ± 0.05	w		
	d-spacings (Å) 11.8 \pm 0.2 10.2 \pm 0.2 7.2 \pm 0.15 4.2 \pm 0.08 3.9 \pm 0.08 3.6 \pm 0.06 3.1 \pm 0.05		

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- Zeolite ZSM-48 can also be identified, in terms of mole ratios of oxides and in the anhydrous state, as follows:
- 30 $(0.1 \text{ to } 4)R_2O:(0.01 \text{ to } 2)M_{2/n}O:(0 \text{ to } 0.5)Al_2O_3:(100)SiO_2$

synthesized and in the anhydrous state, in terms of mole ratios of oxides, is as follows:

- 3 (0.05-2.0)R₂O:(0.1-2.0)M₂O:Al₂O₃:(20-less than 40)SiO₂
- 4 where M is an alkali metal cation and R is an organic nitrogen-containing
- 5 cation, such as an N-lower alkyl-N-N'-isopropyl-imidazolium cation. SSZ-32
- 6 has a mole ratio of silicon oxide to aluminum oxide in the range of 20 to less
- than 40, and has essentially the same X-ray diffraction pattern of ZSM-23.
- 8 Hydroconversion processes using SSZ-32 are disclosed, for example, in U.S.
- 9 Patent Nos. 5,300,210 and in 5,397,454.

ZSM-48 is a crystalline aluminosilicate zeolite which is suitable as a dewaxing catalyst for the present invention. Zeolite ZSM-48 is disclosed in U.S. Patent No. 4,585,747, the entire disclosure of which is incorporated herein by reference for all purposes, and has a characteristic X-ray diffraction pattern as set forth in Table VI.

Table VI

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		145.5 11
17	Interplanar	Relative
18	<u>d-spacings (Å)</u>	Intensity, I/I _o
19	11.8 ± 0.2	S
20	10.2 ± 0.2	w-m
21	7.2 ± 0.15	W
22	4.2 ± 0.08	VS
23	3.9 ± 0.08	VS
24	3.6 ± 0.06	W
25	3.1 ± 0.05	W
26	2.85 ± 0.05	W

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- Zeolite ZSM-48 can also be identified, in terms of mole ratios of oxides and in the anhydrous state, as follows:
- 30 (0.1 to 4) $R_2O:(0.01 \text{ to 2})M_{2/n}O:(0 \text{ to 0.5})Al_2O_3:(100)SiO_2$

other material resistant to the temperatures and other conditions employed in

- 2 organic conversion processes. Such matrix materials include active and
- 3 inactive materials and synthetic or naturally occurring zeolites as well as
- 4 inorganic materials such as clays, silica and metal oxides. Additional porous
- 5 matrix materials include silica, alumina, titania, magnesia and mixtures
- 6 thereof. The matrix can be in the form of a cogel. Alumina and silica-alumina

7 matrix materials are preferred.

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The intermediate pore size molecular sieve is used in admixture with at 8 least one Group VIII metal. Preferably, the Group VIII metal is selected from 9 the group consisting of at least one of platinum and palladium and optionally, 10 other catalytically active metals such as molybdenum, nickel, vanadium, 11 cobalt, tungsten, zinc and mixtures thereof. Most preferably, the Group VIII 12 metal is selected from the group consisting of at least one of platinum and 13 palladium. The amount of metal ranges from about 0.01% to about 10% by 14 weight of the molecular sieve, preferably from about 0.1% to about 5% by 15 weight and more preferably from about 0.2% to about 1% by weight of the 16 molecular sieve. The techniques of introducing catalytically active metals into 17 a molecular sieve are disclosed in the literature, and preexisting metal 18 incorporation techniques and treatment of the molecular sieve to form an 19 active catalyst such as ion exchange, impregnation or occlusion during sieve 20 preparation are suitable for use in the present process. Such techniques are 21 disclosed in U.S. Pat. Nos. 3,236,761; 3,226,339; 3,236,762; 3,620,960; 22 3,373,109; 4,202,996; 4,440,781 and 4,710,485; and in U.S. Application 23 Serial No. 08/728818; the entire disclosures of which are incorporated herein 24 by reference for all purposes. 25

The term "metal" or "active metal" as used herein means one or more metals in the elemental state or in some form such as sulfide, oxide and mixtures thereof. Regardless of the state in which the metallic component actually exists, the concentrations are computed as if they existed in the elemental state.

other material resistant to the temperatures and other conditions employed in organic conversion processes. Such matrix materials include active and inactive materials and synthetic or naturally occurring zeolites as well as inorganic materials such as clays, silica and metal oxides. Additional porous matrix materials include silica, alumina, titania, magnesia and mixtures thereof. The matrix can be in the form of a cogel. Alumina and silica-alumina matrix materials are preferred.

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The intermediate pore size molecular sieve is used in admixture with at least one Group VIII metal. Preferably, the Group VIII metal is selected from the group consisting of at least one of platinum and palladium and optionally, other catalytically active metals such as molybdenum, nickel, vanadium, cobalt, tungsten, zinc and mixtures thereof. Most preferably, the Group VIII metal is selected from the group consisting of at least one of platinum and palladium. The amount of metal ranges from about 0.01% to about 10% by weight of the molecular sieve, preferably from about 0.1% to about 5% by weight and more preferably from about 0.2% to about 1% by weight of the molecular sieve. The techniques of introducing catalytically active metals into a molecular sieve are disclosed in the literature, and preexisting metal incorporation techniques and treatment of the molecular sieve to form an active catalyst such as ion exchange, impregnation or occlusion during sieve preparation are suitable for use in the present process. Such techniques are disclosed in U.S. Pat. Nos. 3,236,761; 3,226,339; 3,236,762; 3,620,960; 3,373,109; 4,202,996; 4,440,781 and 4,710,485; and in U.S. Application Serial No. 08/728818; the entire disclosures of which are incorporated herein by reference for all purposes.

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H₂/kg oil), preferably from about 1,000 to about 10,000 SCF/bbl (151-1510 std 1

- liters H₂/kg oil). Generally, hydrogen will be separated from the product and 2
- recycled to the reaction zone. Strong acidity may also be reduced by 3
- introducing nitrogen compounds, e.g., NH₃ or organic nitrogen compounds, 4
- into the feed; however, the total nitrogen content should be less than 50 ppm, 5
- 6 preferably less than 10 ppm.

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In the dewaxing process using the catalyst of the present invention, the pour point of the isomerized product is lower than the pour point of the waxy feed to the dewaxing process. For oils of commercial interest, the pour point of the oil is generally below about 10°C, and often below 0°C. While a low pour point is desired in the product from the isomerization step, excessive isomerization has a detrimental effect on product viscosity index, as described hereinbefore. The wax content of the isomerized oil is between about 1% and about 40%, preferably between about 3% and about 20%, of the wax content of the waxy feed. The isomerization step, then preferentially removes between about 60% and about 99% by weight of the wax contained in the waxy feedstock. Thus, the pour point of the isomerized product, while being substantially lower than the pour point of the feed to the isomerization process, will be at least about 6°C, and more usually at least about 12°C above the target pour point set for the finished lubricating oil base stock. The 20 viscosity index of the isomerized product will be generally above about 140 and preferably above about 150. With some products, a viscosity index of 22 23 160 or above is possible.

The wax content of the oil set forth herein is determined from a conventional solvent dewaxing method. An example method is as follows:

300 g of oil is diluted 50/50 with a 4:1 mixture of methyl ethyl ketone and toluene which is cooled to -20°C in a refrigerator. The mixture is filtered through a Coors funnel at -15 °C. using Whatman No. 3 filter paper. The wax is removed from the filter and placed in a tared 2 liter flask. The solvent is removed on a hot plate and the wax weighed.

1 H₂/kg oil), preferably from about 1,000 to about 10,000 SCF/bbl (151-1510 std

- 2 liters H₂/kg oil). Generally, hydrogen will be separated from the product and
- 3 recycled to the reaction zone. Strong acidity may also be reduced by
- 4 introducing nitrogen compounds, e.g., NH₃ or organic nitrogen compounds,
- 5 into the feed; however, the total nitrogen content should be less than 50 ppm,
- 6 preferably less than 10 ppm.

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In the dewaxing process using the catalyst of the present invention, the 7 pour point of the isomerized product is lower than the pour point of the waxy 8 feed to the dewaxing process. For oils of commercial interest, the pour point 9 of the oil is generally below about 10°C, and often below 0°C. While a low 10 pour point is desired in the product from the isomerization step, excessive 11 isomerization has a detrimental effect on product viscosity index, as 12 described hereinbefore. The wax content of the isomerized oil is between 13 about 1% and about 40%, preferably between about 3% and about 20%, of 14 the wax content of the waxy feed. The isomerization step, then preferentially 15 removes between about 60% and about 99% by weight of the wax contained 16 in the waxy feedstock. Thus, the pour point of the isomerized product, while 17 being substantially lower than the pour point of the feed to the isomerization 18 process, will be at least about 6°C, and more usually at least about 12°C 19 above the target pour point set for the finished lubricating oil base stock. The 20 viscosity index of the isomerized product will be generally above about 140 21 and preferably above about 150. With some products, a viscosity index of 22 23 160 or above is possible.

The wax content of the oil set forth herein is determined from a conventional solvent dewaxing method. An example method is as follows:

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described, for example, in U.S. Patent No. 5,158,665, the entire disclosure of which is already incorporated by reference.

It may further be desired to hydrofinish the dewaxed oil in a mild hydrogenation process to produce more stable lubrication oils. The hydrofinishing can be conventionally carried out in the presence of a metallic hydrogenation catalyst, for example, platinum on alumina. The hydrofinishing can be carried out at a temperature of from about 190°C to about 340°C and a pressure of from about 400 psig to about 3000 psig (2.76-20.7 MPa). A description of a typical hydrofinishing process and catalyst which is useful in the present process is taught in U.S. Patent No. 5,158,665. Hydrofinishing in this manner is also described in U.S. Pat. 3,852,207, both of which are incorporated herein by reference for all purposes.

The present process is suitable for preparing very high viscosity index lubricating oil base stocks having a wide range of viscosities, including base stocks having a viscosity, measured at 100°C, of 10 cSt or higher. These base oils have a viscosity index of at least about 140 (preferably at least about 150 and more preferably at least about 160), and a pour point of less than or equal to about -10°C (preferably less than or equal to about -20°C, and more preferably less than or equal to about -30°C). A particularly important base oil prepared in the present process has a viscosity, measured at 100°C, of about 3 cSt or less, preferably at least about 150, and more preferably at least about 140, preferably at least about 150, and more preferably at least about 160. This relatively light oil prepared in the present process has a viscosity index higher than that produced even in synthetic oils having a viscosity, measured at 100°C, of about 3 cSt or less.

26 <u>EXAMPLES</u>

Comparative Example A

Tetracosane (n-C₂₄, purchased from Aldrich), which had a pour point of +50 C and a viscosity at 100 C of about 2.5 cSt, was isomerized over SM-3 impregnated with 0.5 wt% Pt. The catalyst was pelleted, then crushed to 24-

described, for example, in U.S. Patent No. 5,158,665, the entire disclosure of which is already incorporated by reference.

It may further be desired to hydrofinish the dewaxed oil in a mild hydrogenation process to produce more stable lubrication oils. The hydrofinishing can be conventionally carried out in the presence of a metallic hydrogenation catalyst, for example, platinum on alumina. The hydrofinishing can be carried out at a temperature of from about 190°C to about 340°C and a pressure of from about 400 psig to about 3000 psig (2.76-20.7 MPa). A description of a typical hydrofinishing process and catalyst which is useful in the present process is taught in U.S. Patent No. 5,158,665. Hydrofinishing in this manner is also described in U.S. Pat. 3,852,207, both of which are incorporated herein by reference for all purposes.

The present process is suitable for preparing very high viscosity index lubricating oil base stocks having a wide range of viscosities, including base stocks having a viscosity, measured at 100°C, of 10 cSt or higher. These base oils have a viscosity index of at least about 140 (preferably at least about 150 and more preferably at least about 160), and a pour point of less than or equal to about -10°C (preferably less than or equal to about -20°C, and more preferably less than or equal to about -30°C). A particularly important base oil prepared in the present process has a viscosity, measured at 100°C, of about 3 cSt or less, preferably less than about 3 cSt, and a viscosity index of at least about 140, preferably at least about 150, and more preferably at least about 160. This relatively light oil prepared in the present process has a viscosity index higher than that produced even in synthetic oils having a viscosity, measured at 100°C, of about 3 cSt or less.

EXAMPLES PROPERTY NAMED IN COLUMN 1

Comparative Example A

Tetracosane (n-C₂₄, purchased from Aldrich), which had a pour point of +50 C and a viscosity at 100 C of about 2.5 cSt, was isomerized over SM-3 impregnated with 0.5 wt% Pt. The catalyst was pelleted, then crushed to 24-

1 2 **TABLE VII** ISOMERIZATION OF n-C24 OVER Pt/SM-3 AT 3 1000 PSIG (6.99 MPa), 0.6 hr⁻¹ LHSV, 4 AND 6.7 MSCF/BBL H2 (1010 std liters H2/kg oil) 5 6 7 Comparative Example A Example 1 332 324 8 Temperature, °C 321 99.1 99.6 95.1 9 n-C₂₄ Conversion, wt% 10 11 Yield, Wt% 0.2 12 C4-0.5 0.9 0.5 2.3 13 C5-82 °C 1.9 1.7 14 2.8 3.2 82-177 °C 4.3 15 8.2 12.3 177-316°C 93.3 316 °C+ 86.6 81.3 16 17 92.1 18 87.4 82.2 316°C+ Distillation Yield, wt% 19 20 No No Yes Solvent Dewax 65.6 21 Oil, wt% 32.4 22 Wax, wt% +20 23 Pour Point Before SDW, °C 24 61.2 81.3 25 86.6 316°C+ Lube Yield, wt% 26 27 316 °C+ Lube Inspections -29 -25 28 Pour Point, °C -15 -9 -1 -8 29 Cloud Point, °C Viscosity, 40 °C, cSt 8.313 8.372 8.636 30 2.556 31 100 °C, cSt 2.579 2.507 148 32 VI 137 132 33 1.63 1.83 1.97 34 Avg. Branches/Molecule 35 36 Simulated Distillation, LV%, °C 294/357 304/369 37 277/358 St/5 374/382 38 30/50 368/379 368/379 385 39 50 384 384 388/391 388/391 40 70/90 388/392 392/398 393/394 41 95/EP 392/394

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2		TABLE VI	1						
3	ISOMERIZATION OF n-C ₂₄ OVER Pt/SM-3 AT								
4	1000 PSIG (6.99 MPa), 0.6 hr ⁻¹ LHSV,								
5	AND 6.7 MSCF/BBL H ₂ (1010 std liters H ₂ /kg oil)								
5 6	7 (14D 0.1 MOO! 1DDE 112 (10 10 010 mo.0 1.2mg 0m)								
7		Comparative	Example A	Example 1					
8	Temperature, °C	321	332	324					
9	n-C ₂₄ Conversion, wt%	99.1	99.6	95.1					
10	•								
11	Yield, Wt%								
12	C4-	0.5	0.9	0.2					
13	C5-82 °C	1.9	2.3	0.5					
14	82-177 °C	2.8	3.2	1.7					
15	177-316°C	8.2	12.3	4.3					
16	316 °C+	86.6	81.3	93.3					
17				20.4					
18	316°C+ Distillation Yield, wt%	87.4	82.2	92.1					
19				V = -					
20	Solvent Dewax	No	No	Yes					
21	Oil, wt%			65.6					
22	Wax, wt%			32.4					
23	Pour Point Before SDW, °C			+20					
24			04.0	61.2					
25	316°C+ Lube Yield, wt%	86.6	81.3	01.2					
26									
27	316 °C+ Lube Inspections			00					
28	Pour Point, °C	-15	-25	-29					
29	Cloud Point, °C	-1	-8	-9					
30	Viscosity, 40 °C, cSt	8.636	8.372	8.313					
31	100 °C, cSt	2.579	2.507	2.556					
32	VI	137	132	148					
33			4.07	4.60					
34	Avg. Branches/Molecule	1.83	1.97	1.63					
35		•							
36	Simulated Distillation, LV%, °C		004/057	204/260					
37	St/5	277/358	294/357	304/369 374/382					
38	30/50	368/379	368/379	374/302					
39	50	384	384	388/391					
40	70/90	388/392	388/391 393/394	392/398					
41	95/EP	392/394	J3J/J34	5921590					

1 2 3 4 5	TABLE IX ISOMERIZATION OF HEAVY NEUTRAL SLACK WAX AT 0.5 hr ⁻¹ LHSV, 1000 PSIG (6.99 MPa), AND 8 MSCF/BBL H ₂ (1210 std liters H ₂ /kg oil) OVER Pt/SM-3 CATALYST				
6 7	Comparative Example B Example 2				
8	Temperature, °C	349	332		
10 11	343°F+ Conversion, wt%	27.4 100	21.9 84.1		
12 13	Wax Conversion, wt% Selectivity to Lube, wt%	67.4	72.6		
14 15	Pour Point before SDW, °C	C	0		
16 17	Solvent Dewax	No .	Yes		
18 19	Oil, wt% Wax, wt%		86.2 13.8		
20 21	650 F+ Lube Yield, wt%	67.4	61.1		
22 23	Pour Point, °C	-12	-18 -17		
24 25	Cloud Point, °C	+9	37.50		
26 27	Viscosity, 40 °C, cSt 100 °C, cSt	41.42 7.367	6.836		
28 29	VI	144	143		
30 31	Simulated Distillation, LV% St/5	%, °C 193/357	226/358		
32	30/50	378/425	377/419		
33	50	464	456		
34	70/90	511/585	500/579		
35	95/EP	617 <i>[</i> 717	629/747		
36					
37	Comparative Example C				
38	An SM-3 catalyst similar to that of Comparative Example B was used				
39		to isomerize a hydrotreated 4.5 cSt slack wax (Table X) at 0.5 hr ⁻¹ LHSV, 800			
40	psig total pressure (5.61 MPa), and 3 MSCF/bbl H ₂ (454 std liters H ₂ /kg oil).				
41	Results are given in Table XI, showing a 140 VI at a pour point of -7°C.				

1 2 3 4 5	TABLE IX ISOMERIZATION OF HEAVY NEUTRAL SLACK WAX AT 0.5 hr 1 LHSV, 1000 PSIG (6.99 MPa), AND 8 MSCF/BBL H ₂ (1210 std liters H ₂ /kg oil) OVER Pt/SM-3 CATALYST			
6 7	Comparative Example B Example 2			
8 9	Temperature, °C	349	332	
10 11	343°F+ Conversion, wt%	27.4	21.9	
12	Wax Conversion, wt%	100	84.1	
13	Selectivity to Lube, wt%	67.4	72.6	
14	Ociobatity to Edbol 11179			
15	Pour Point before SDW,	°C	0	
16	Four Fourt Deloie ODVV,	J		
17	Solvent Dewax	No .	Yes	
	Oil, wt%		86.2	
18	Wax, wt%		13.8	
19	VVax, Wt76			
20	GEO EL Luba Viald 144%	67.4	61.1	
21	650 F+ Lube Yield, wt%	07.4		
22	Dour Doint °C	-12	-18	
23	Pour Point, °C	+9	-17	
24	Cloud Point, °C			
25	\" \ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\	41.42	37.50	
26	Viscosity, 40 °C, cSt		6.836	
27	100 °C, cSt	7.367	143	
28	VI	144	145	
29		(0/ 90		
30	Simulated Distillation, L\	/%, °C	226/358	
31	St/5	193/357	377/419	
32	30/50	378/425		
33	50	464	456	
34	70/90	511/585	500/579	
35	95/EP	617 <i>/</i> 717	629/747	
36				
		Comparative Example	e C	
37		Comparative Example C		
38	An SM-3 catalyst similar to that of Comparative Example B was used			
39	to isomerize a hydrotreated 4.5 cSt slack wax (Table X) at 0.5 hr ⁻¹ LHSV, 800			
40	psig total pressure (5.61 MPa), and 3 MSCF/bbl H ₂ (454 std liters H ₂ /kg oil).			
41	Results are given in Table XI, showing a 140 VI at a pour point of –7°C.			

1 2 3 4	TABLE XI ISOMERIZATION OF HYDROTREATED SLACK WAX AT 0.5 hr ⁻¹ LHSV AND 3 MSCF/BBL H ₂ (450 std liters H ₂ /kg oil) OVER Pt/SM-3 CATALYST			
5 6		Comparative Example C	Example 3	
7	Temperature, °C	327	327	
8	Pressure, MPa	5.61	7.68	
9				
10	Conversion <371°C, w	t% 28.9	23.7	
11				
12	Yields, Wt%			
13	C4-	2.2	2.0	
14	C5-82 °C	3.8	3.3	
15	180-371 °C	31.7	27.8	
16	371°C+	62.8	67.3	
17				
18	371°C+ Yield, Wt%	62.6	66.8	
19			_	
20	Pour Point before SDV	N, °C	-3	
21				
22	Solvent Dewax	No	Yes	
23	Oil, wt%		96	
24	Wax, wt%		4	
25			64	
26	371°C+ Lube Yield, w	t% 62.6	64	
27	D D: 4 10	7	-14	
28	Pour Point, °C	- 7	-14 -11	
29	Cloud Point, °C	-4	-11	
30	\financity 40 °C act	22.0	21.98	
31 32	Viscosity, 40 °C, cSt 100 °C, cSt	4.746	4.785	
32 33	100 C, cst VI	140	144	
33 34	VI	140	1-1-1	
3 4 35	Simulated Distillation, LV%, °C			
36	ST/5	287/368	294/371	
37	30/50	436/452	738/454	
38	95/99	486/501	488/502	
39	00/00			
40				

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1 2 3 4	TABLE XI ISOMERIZATION OF HYDROTREATED SLACK WAX AT 0.5 hr ⁻¹ LHSV AND 3 MSCF/BBL H ₂ (450 std liters H ₂ /kg oil) OVER Pt/SM-3 CATALYST			
5		comparative Example C	Example 3	
6	Temperature, °C	327	327	
7 8	Pressure, MPa	5.61	7.68	
	Plessuie, wii a	5.5 .		
9	Conversion <371°C, wt	% 28.9	23.7	
10	Conversion 371 0, we	70 20.0		
11 12	Yields, Wt%			
	C4-	2.2	2.0	
13	C5-82 °C	3.8	3.3	
14	180-371 °C	31.7	27.8	
15 16	371°C+	62.8	67.3	
16	371 0+	02.0		
17	371°C+ Yield, Wt%	62.6	66.8	
18	3/1 C+ Held, VVI/0	02.0		
19	Pour Point before SDV	. °C	-3	
20	Pour Point before 3DV	ν, Ο		
21	Calvert Dower	No	Yes	
22	Solvent Dewax	140	96	
23	Oil, wt%		4	
24	Wax, wt%		•	
25	ozaso i uka Viold ut	% 62.6	64	
26	371°C+ Lube Yield, wt	76 02.0	•	
27	D. Deint 90	-7	-14	
28	Pour Point, °C	- <i>t</i> -4	-11	
29	Cloud Point, °C	7	• •	
30	10 00 00	22.0	21.98	
31	Viscosity, 40 °C, cSt	4.746	4.785	
32	100 °C, cSt	140	144	
33	VI	140	• • • •	
34	20 1 1 D' (11 1 2 - 1 1 1 0 / 20			
35	Simulated Distillation,	287/368	294/371	
36	ST/5	436/452	738/454	
37	30/50	486/501	488/502	
38	95/99	700/301	.30.00	
39				
40				

1							
2	TABLE XII						
3	ISOMERIZ	ZATION OF n-0	C ₂₄				
4	AT 1000 PSIG (6	.99 MPa), 0.6 I	nr ⁻¹ LHSV,				
5	AND 6.7 MSCF/BBL	H ₂ (1010 std li	ters h2/kg oil)				
6	OVER Pt/S	SZ-32 CATAL	YST Ü				
6 7							
8		Comparative	e Example D	Example 4			
9	Temperature, °C	307	324	310			
10	n-C ₂₄ Conversion, wt%	98.9	99.8	87.9			
11	24 5511111111111111111111111111111111111						
12	Yields, Wt%						
13	C1-C2	0.3	0.4	0.3			
14	C3-C4	4.7	5.4	1.8			
15	C5-82 °C	7.4	8.4	2.7			
16	82-177 °C	11.9	12.0	2.8			
17	177-316 °C	12.2	14.8	8.8			
18	316 °C+	63.5	59.0	82.7			
19							
20	316 °C+ Distillation Yield, W	t% 64.4	68.5	88.9			
21	•						
22	Solvent Dewax	No	No	Yes			
23	Oil, Wt%			86.1			
24	Wax, Wt%			11.5			
25							
26	Pour Point before SDW, °C			+4			
27							
28	316 °C+ Lube Yield, Wt%	63.5	59.0	52.9			
29							
	040.80 : 1 :- 1 1						
30	316 °C+ Lube Inspections			<u> </u>			
31	Pour Point, °C	-9	-33	-21			
32	Cloud Point, C	+2	-13	-7			
33				7.000			
34	Viscosity, 40 °C, cSt	8.028	6.414	7.669			
35	100 °C, cSt	2.506	2.121	2.445			
36	VI	152	143	156			
37		4.00					
38	Avg. Branches/Molecule	1.60					
39							
40	Simulated Distillation, LV%,		4501040	249/204			
41	St/5	273/333	156/240	218/294 373/385			
42	30/50	371/383	278/373	373/300			
43	50 70/00	387	380	391/394			
44	70/90	390/393	383/387	394/394			
45	95/EP	393/395	388/391	354/354			

Comparative Example E

A boron-Beta zeolite was prepared according to Example 18 of US Patent No. 5,558,851. This zeolite, which had a SiO₂/B₂O₃ mole ratio of about 60, was NH4-exchanged and then impregnated with 0.5 wt% Pt. The catalyst was pelleted and crushed to 24-42 mesh (0.35-0.70 mm). After presulfiding with H₂S, the catalyst was used to isomerize tetracosane at 1000 psig (6.99 MPa), 0.6 hr⁻¹ LHSV, and 6.7 MSCF/bbl H₂ (1010 std liters H₂/kg oil) to a pour point of +16°C, then solvent dewaxed to a pour point of –18°C. The viscosity index after solvent dewaxing was considerably lower than for the catalysts of this invention (Table XIII).

Comparative Example F

0.5 wt% Pt was impregnated on an amorphous cogelled SiO2-alumina base extrudate (31 wt% SiO2, 69 wt% Al2O3). The catalyst was crushed to 24-42 mesh (0.35-0.70 mm) for testing. After pre-sulfiding with H₂S, it was used to isomerize tetracosane at 1000 psig (6.99 MPa), 0.6 LHSV, and 6.7 MSCF/bbl H₂ (1010 std liters H₂/kg oil) to a pour point of +22°C, then solvent dewaxed to a pour point of -15°C. The viscosity index after solvent dewaxing was considerably lower than for the catalysts of this invention (Table XIII and Figure 2). In addition, the isomerized and solvent dewaxed oil had a much higher average number of branches per molecule.

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1 2 **TABLE XIII** ISOMERIZATION OF n-C $_{24}$ AT 1000 PSIG (6.99 MPa), 0.6 hr $^{-1}$ LHSV, 3 4 AND 6.7 MSCF/BBL H₂ (1010 std liters H₂/kg oil) 5 6 Example 1 7 Comparative Comparative Example F Example E 8 Pt/SiO2-Al2O3 Pt/SM-3 Pt/B-Beta 9 Catalyst 10 329 324 319 11 Temperature, °C 95.1 92.4 n-C24 Conversion, Wt% 95.2 12 13 14 Yields, Wt% 0.2 0.3 2.8 15 C4-0.5 1.3 5.4 C5-82 °C 16 1.7 82-177 °C 7.3 2.0 17 6.7 4.3 177-316 °C 16.6 18 93.3 89.7 67.9 19 316 °C+ 20 92.1 90.3 69.0 21 316 °C+ Dist. Yield, Wt% 22 Yes Yes Yes 23 **Solvent Dewax** 65.6 86.1 86.4 Oil, wt% 24 11.5 32.4 13.1 25 Wax, wt% +22 +20 +16 Pour Point before SDW, °C 26 27 61.2 316 °C+ Lube Yield, Wt% 58.7 77.2 28 29 30 316 °C+ Lube Inspections -29 -15 Pour Point, °C -18 31 -9 Cloud Point, °C -13 -11 32 33 8.313 8.354 8.364 Viscosity, 40 °C, cSt 34 2.556 2.481 100 °C, cSt 2.517 35 148 126 VΙ 136 36 37 1.63 1.86 2.02 38 Avg. Branches/Molecule 39 40 Simulated Dist., LV%,°C 304/369 316/360 41 St/5 298/343 30/50 364/375 365/375 374/382 42 385 375 381 43 50 388/391 385/390 44 70/90 385/389 390/392 391/392 392/398

95/EP

1 WHAT IS CLAIMED IS:

A process for preparing an oil suitable for use as a lubricating oil base

- 3 stock and having a viscosity index of greater than 140 and a target pour
- 4 point of less than or equal to -10°C comprising:
- 5 a) contacting a waxy feed over a catalyst comprising a molecular sieve
- 6 having 1-D pores with a pore diameter of between about 5.0 Å and
- 7 about 7.0 Å, and at least one Group VIII metal, at a pressure of from
- 8 about 15 psig (103 kPa) to about 2500 psig (13.8 MPa) to produce an
- g isomerized oil having a pour point of at least 6°C above a target pour
- 10 point; and
- b) solvent dewaxing the isomerized oil to produce a lubricating oil base
- stock having the target pour point and a viscosity index of greater than
- 13 about 140.
- 14 2. The process according to Claim 1 for preparing a lubricating oil base
- stock having a target pour point of less than about -20°C.
- 16 3. The process according to Claim 1 for preparing a lubricating oil base
- stock having a viscosity index of greater than 150.
- 18 4. The process according to Claim 1wherein the waxy feed contains more
- 19 than about 50% wax.
- The process according to claim 4 wherein the waxy feed contains more
- than about 80% wax.
- 22 6. The process according to Claim 1 wherein the waxy feed contains more
- than about 70% paraffinic carbon.
- 24 7. The process according to Claim 1 wherein the waxy feed is selected from
- 25 the group consisting of synthetic oils and waxes such as those by
- 26 Fischer-Tropsch synthesis, high pour point polyalphaolefins, foots oils,
- 27 normal alpha olefin waxes, slack waxes, deoiled waxes and
- 28 microcrystalline waxes.

1 8. The process according to Claim 4 wherein the waxy feed is selected from

- the group consisting of synthetic oils and waxes such as those by
- 3 Fischer-Tropsch synthesis, high pour point polyalphaolefins, foots oils,
- 4 normal alpha olefin waxes, slack waxes, deoiled waxes and
- 5 microcrystalline waxes.
- 6 9. The process according to Claim 1 wherein the isomerized oil has a pour
- 7 point of greater than about 0°C.
- 8 10. The process according to Claim 1 wherein between about 60% and about
- 9 99% by weight of the wax contained in the waxy feedstock is removed in
- 10 step a).
- 11 11. The process according to Claim 1 wherein the medium pore molecular
- sieve has 1-D pores having a minor axis between about 3.9Å and about
- 13 4.8Å and a major axes between about 5.4Å and about 7.0Å.
- 14 12. The process according to Claim 1 wherein the medium pore molecular
- sieve is selected from the group consisting of SAPO-11, SAPO-31 and
- 16 SAPO-41.
- 17 13. The process according to Claim 12 wherein the medium pore molecular
- 18 sieve is SM-3.
- 19 14. The process according to Claim 1 wherein the medium pore molecular
- sieve is selected from the group consisting of ZSM-22, ZSM-23, ZSM-35
- 21 and SSZ-32.
- 22 15. The process according to Claim 14 wherein the medium pore molecular
- 23 sieve is SSZ-32.
- 24 16. The process according to Claim 1 wherein the medium pore molecular
- sieve is ZSM-48.
- 26 17. The process according to Claim 1 wherein the hydrogenation component
- is a Group VIII metal selected from the group consisting of platinum,
- 28 palladium or mixtures thereof.

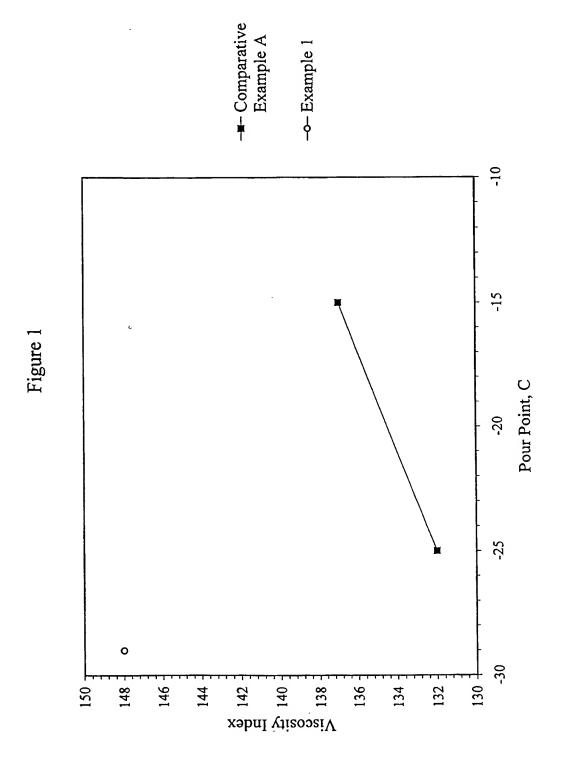
1 18. The process according to claim 17 wherein the catalyst contains from about 0.2% to about 1% by weight of the hydrogenation component.

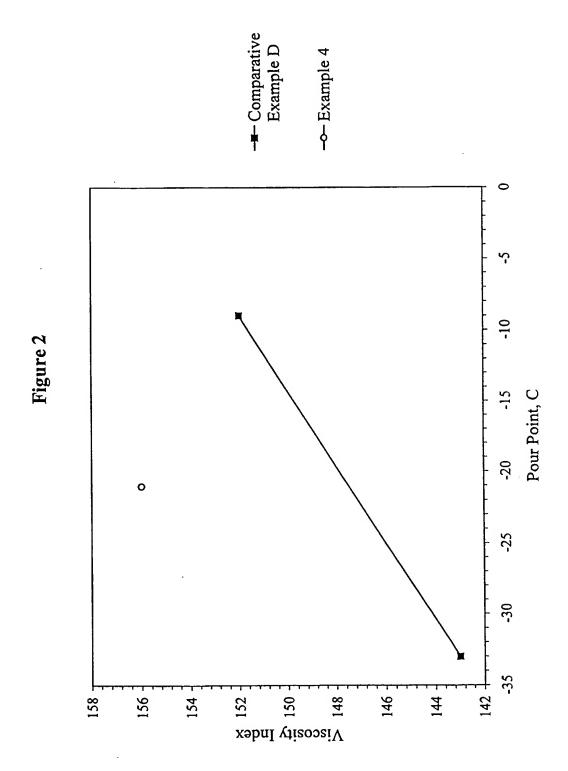
- 3 19. The process of claim 1 wherein the catalyst comprising the molecular
- 4 sieve has sufficient isomerization selectivity such that, when contacting a
- 5 n-C₂₄ feed at a total pressure of 1000 psig (6.99 MPa), hydrogen flow
- equivalent to 6.7 MSCF/bbl (1010 std liters H₂/kg oil), and a feed rate
- 7 equivalent to 0.6 hr⁻¹ LHSV with the catalyst, to produce a 316°C+
- 8 dewaxed product having a pour point of about +20°C and solvent
- 9 dewaxing the dewaxed product to a pour point of -15°C or below, an
- isomerized product having a branching index of less than about 1.75 is
- 11 formed.
- 12 20. A process for preparing an oil suitable for use as a lubricating oil base
- 13 stock comprising:
- a) contacting a waxy feed over a catalyst comprising a molecular sieve
- having 1-D pores with a pore diameter of between about 5.0 Å and
- about 7.0 Å, and at least one Group VIII metal, at a pressure of from
- about 15 psig (103 kPa) to about 2500 psig (13.8 MPa) to produce an
- isomerized oil having a pour point of greater than about 0°C; and
- b) solvent dewaxing the isomerized oil to produce a lubricating oil base
- 20 stock having a pour point of less than or equal to -10°C, a viscosity
- 21 index of greater than about 140 and a viscosity, measured at 100°C, of
- 22 about 3 cSt or less.
- 23 21. The process according to Claim 20 wherein the viscosity of the lubricating
- oil base stock, measured at 100°C, is less than about 3 cSt and the pour
- point is less than or equal to -20°C.
- 26 22. The process according to Claim 20 wherein the viscosity index of the
- lubricating oil base stock is greater than 150 and the pour point is less
- 28 than –20°C.
- 29 23. The process according to Claim 20 wherein the molecular sieve is
- 30 SSZ-32.

1 24. The process according to Claim 20 wherein the molecular sieve is SM-3.

- 2 25. A lubricating oil base stock having a viscosity index of at least about 140,
- a pour point of less than or equal to -10°C, and a viscosity, measured at
- 4 100°C, of about 3 cSt or less.
- 5 26. The lubricating oil base stock of Claim 25 having a viscosity index of at
- 6 least about 150 and a pour point of less than or equal to -20°C.

7





INTERNATIONAL SEARCH REPORT

Ir. ational Application No PCT/US 99/02121

			101700 33702121				
A. CLASSI IPC 6	FICATION OF SUBJECT MATTER C10G67/04 C10G45/64						
According to	o International Patent Classification (IPC) or to both national classific	ation and IPC					
	SEARCHED						
Minimum do	cumentation searched (classification system followed by classification ${\tt C10G}$	on symbols)					
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched							
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)							
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT						
Category *	Citation of document, with indication, where appropriate, of the rel	evant passages	Relevant to claim No.				
X	US 4 919 788 A (CHEN NAI Y ET AL 24 April 1990	_)	1-10, 17-22, 25,26				
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	-	-/					
:							
Further documents are listed in the continuation of box C. X Patent family members are listed in annex.							
	tegories of cited documents :	"T" later document publis	shed after the international filing date				
"A" document defining the general state of the art which is not considered to be of particular relevance or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention							
"E" earlier document but published on or after the international filing date "L" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone							
which is cited to establish the publication date of another citation or other special reason (as specified) "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document referring to an oral disclosure, use, exhibition or							
"P" docume	other means ments, such combination being obvious to a person skilled						
Date of the	actual completion of the international search	Date of mailing of th	ne international search report				
20	6 April 1999	06/05/19	999				
Name and mailing address of the ISA Authorized officer Authorized officer							
European Patent Office, P.B. 5818 Patentiaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Michiels, P					

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INTERNATIONAL SEARCH REPORT

tr atlanal Application No PCT/US 99/02121

	tion) DOCUMENTS CONSIDERED TO BE RELEVANT	Relevant to claim No.	
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